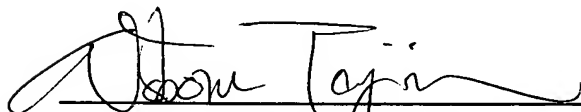


VERIFICATION OF TRANSLATION

I, NOBORU TAJIME, a Japanese Patent Attorney registered No.9558, having my business office at Room No.201, New-Well-Ikuta Bldg., 26-28, Mita 1-chome, Tama-ku, Kawasaki-shi, Kanagawa, 214-0034 JAPAN, hereby declare that I am a translator of the specification, claims and abstract of The International Application No. PCT/JP2004/008024 and that the following is a true translation thereof to the best of knowledge and belief.

Signed this 15th day of December, 2005


Noboru TAJIME

DESCRIPTION

INSULATION COATED CONDUCTIVE PARTICLE

TECHNICAL FIELD

5 [0001]

The present invention relates to an insulation coated conductive particle used in an anisotropic conductive adhesive.

BACKGROUND ART

10 [0002]

Insulation coated conductive particles, formed by coating the surface of conductive particles with a thermoplastic insulating resin, are widely used in anisotropic conductive adhesives. In such uses, the coating is used to prevent occurrence of short circuits between the conductive particles. Examples of conductive particles include metal particles such as nickel particles, and metal plated particles each having a metal plating layer formed on the surface of a resin particle.

20 Patent Document 1: Japanese Patent Application Laid-Open No. Hei 5-217617

Patent Document 2: Japanese Patent Application Laid-Open No. Hei 5-70750

[0003]

25 However, when a film or a paste of the anisotropic

conductive adhesive is manufactured using the
aforementioned insulation coated conductive particles, the
insulating resin layer that coats the conductive particles
swells, dissolves, or is deformed in the solvents
5 sometimes used in the manufacturing process. In those
cases, a conduction reliability of the anisotropic
conductive adhesive is adversely affected.

[0004]

In order to improve a solvent resistance of the
10 insulating resin layer, the insulating resin layer is
considered to be formed from a thermosetting insulating
resin composition. However, when the insulating resin
layer is too hard, it is not possible to remove
sufficiently the insulating resin layer from a region
15 between opposed electrodes that are to be connected to
each other. Thus, the required conduction between the
electrodes cannot be reliably obtained.

[0005]

It is an object of the present invention to provide
20 insulation coated conductive particle exhibiting excellent
solvent resistance as well as providing reliable
conduction properties, when used in an anisotropic
conductive adhesive.

25 DISCLOSURE OF THE INVENTION

[0006]

The inventor of the present invention found that it was possible to improve the solvent-resistance and conduction reliability of insulation coated conductive particles when an insulating resin having a carboxyl group was formed as an insulating resin layer on the surface of a conductive particle, and then the insulating resin layer was subjected to a surface treatment by a polyfunctional aziridine compound having two or more aziridine groups such that there was reaction between the carboxyl and aziridine groups. Based on this, the inventor arrived at the present invention.

[0007]

That is, the present invention provides an insulation coated conductive particle in which the surface of a conductive particle is coated with an insulating resin layer formed of an insulating resin having a carboxyl group, wherein the insulating resin layer is surface-treated with a polyfunctional aziridine compound.

[0008]

Moreover, the present invention provides a method for fabricating an insulation coated conductive particle, which method includes the step of performing a surface treatment with a polyfunctional aziridine compound on an insulating resin layer that is formed of an insulating

resin having a carboxyl group, and coats the surface of a conductive particle.

[0009]

Furthermore, the present invention provides an
5 anisotropic conductive adhesive in which the
aforementioned insulation coated conductive particles are
dispersed in an insulating adhesive.

[0010]

According to the present invention, it is possible
10 to provide both an excellent solvent-resistance and a
conduction reliability to an insulation coated conductive
particle that is suitable for use as a conductive particle
in an anisotropic conductive adhesive.

15 BEST MODE FOR CARRYING OUT THE INVENTION

[0011]

An insulation coated conductive particle of the
present invention is formed by coating the surface of a
conductive particle with an insulating resin layer.

20 [0012]

In the present invention, an insulating resin having
a carboxyl group is used as the resin for the insulating
resin layer that coats the conductive particle. Thus,
adhesion between the conductive particle and the
25 insulating resin layer can be improved. An example of the

insulating resin having a carboxyl group is an insulating resin having a monomer having a carboxyl group -

preferably an acrylic acid monomer unit or a methacrylic acid monomer unit. Specific examples include acrylic

5 acid-styrene copolymer (PP-2000S, Dainippon Ink and Chemicals Inc., acid number 5 mgKOH/g or less), carboxylic acid modified styrene-divinylbenzene copolymer (SX8742A manufactured by JSR Corporation, an acid value of about 3.5 mgKOH/g), and other copolymers.

10 [0013]

When the carboxyl amount (acid value) in the insulating resin is too small, the solvent resistance is insufficient. On the other hand, when the carboxyl amount in the insulating resin is too large, the cross-linking

15 density is excessively high and therefore conduction reliability is impaired. Thus, the carboxyl amount in the insulating resin is preferably in the range from 0.1 to 50 mgKOH/g, and more preferably in the range from 0.5 to 5 mgKOH/g.

20 [0014]

The thickness of the insulating resin layer is preferably in the range from 0.01 to 1 μm , and more preferably, in the range from 0.1 to 0.5 μm . This is because insulation provided by the insulating resin layer

25 is insufficient when the layer is too thin, and the

conduction properties thereof are low when the layer is too thick.

[0015]

As described above, in the insulation coated
5 conductive particle of the present invention, the surface
of the conductive particle is coated with an insulating
resin layer formed of an insulating resin having a
carboxyl group. Thus, in order to ensure a sufficient
conduction reliability in the resulting anisotropic
10 conductive adhesive, the insulating resin layer of the
insulation coated conductive particle should be removed
from a region between portions to be connected during
thermal compression bonding. Therefore, the insulating
resin layer should be thermoplastic under the conditions
15 of a thermal treatment. However, the thermoplastic
insulating resin can easily swell in organic solvents and
may be dissolved by organic solvents in some cases. Thus,
such a thermoplastic insulating resin has a problem of the
solvent-resistance. Moreover, a carboxyl group can easily
20 react with the epoxy group of an epoxy resin, these being
generally used as an adhesive component of anisotropic
conductive adhesives. Thus, the carboxyl group may reduce
the storage stability of anisotropic conductive adhesives.
[0016]

25 Therefore, in the present invention, the insulating

resin layer of the insulation coated conductive particle is surface-treated with a polyfunctional aziridine compound having two or more aziridine groups. This surface treatment is performed to cause reaction between the aziridine groups of the polyfunctional aziridine compound and the carboxyl groups of the insulating resin. More specifically, a solution (e.g., an ethanol solution) of the polyfunctional aziridine compound is typically sprayed onto the surface of the insulating resin layer and is then heated and dried at a temperature from 80°C to 140°C. Thus, reaction between the aziridine groups and the carboxyl groups is effected. Alternatively, this reaction may be effected by adding conductive particles coated with the insulating resin into a solution (e.g., an ethanol solution) of the polyfunctional aziridine compound, dispersing those conductive particles while stirring the mixture, and then stirring and heating the mixture at a temperature from 30°C to 80° in that state. As a result, the surface of the insulating resin layer is cross-linked by the aziridine compound. Therefore, the solvent resistance of the insulation coated conductive particle can be improved without damaging a thermoplastic property of the insulating resin layer. Moreover, free carboxyl groups can be eliminated. Thus, the storage stability of an anisotropic conductive adhesive can be improved even if

epoxy resin is used as the adhesive component.

[0017]

Please note that the reaction between the aziridine group and the carboxyl group is widely known (see

5 Encyclopedia of Chemical Technology, vol. 13, pp. 142 to 166 (1984), for example).

[0018]

It is preferable that the number of aziridine groups in the polyfunctional aziridine compound be at least two

10 from a viewpoint of providing cross-linking. Specific examples of the polyfunctional aziridine compound include trimethylolpropane-tri- β -aziridinypropionate, tetramethylolmethane-tri- β -aziridinypropionate, and N,N-hexamethylene-1,6-bis-1-aziridinecarboxamide.

15 Trimethylolpropane-tri- β -aziridinypropionate is more preferable from a viewpoint of reactivity.

[0019]

The amount of polyfunctional aziridine compound to be used is appropriately determined in accordance with the

20 number of aziridine groups in the aziridine compound, the carboxyl equivalent of the insulating resin, the required level of the solvent-resistance, and the like.

[0020]

A conductive particle having the same structure as

25 that used in conventional anisotropic conductive adhesives

can be used as the conductive particle in the insulation coated conductive particle of the present invention.

Examples of such a conductive particle include a solder particle, a nickel particle, a resin particle plated with a metal (e.g., nickel, gold, or the like), and a particle
5 a metal (e.g., nickel, gold, or the like), and a particle formed by coating any of those particles with an insulating material. Among the above particles, a resin particle plated with nickel and gold and which exhibits good conduction reliability may be preferably used.

10 [0021]

When the average particle diameter of the conductive particles used in the present invention is too small, the conduction reliability may deteriorate. On the other hand, when the average particle diameter is too large,
15 insulating properties may be insufficient. Therefore, it is preferable that the average particle diameter be in the range from 2 to 10 μm .

[0022]

The insulation coated conductive particle of the
20 present invention can be fabricated by arranging a polyfunctional aziridine compound on the surface of an insulating resin layer, formed of an insulating resin having a carboxyl group, which coats a conductive particle, and then heating the polyfunctional aziridine compound and
25 the insulating resin layer to cause reaction between the

carboxyl group of the insulating resin and the aziridine group of the aziridine compound. More specifically, the surface of the conductive particle is coated with an insulating resin in a usual manner. Then, a solution
5 (e.g., ethanol solution) of a polyfunctional aziridine compound is sprayed onto the surface of the insulating resin and is dried and heated at a temperature from 80°C to 140°C. In this manner, the reaction between the carboxyl group and the aziridine group can be effected.
10 Alternatively, the above reaction may be effected by adding the conductive particles coated with the insulating resin to the solution of a polyfunctional aziridine compound and then heating the mixture at a temperature from 30°C to 80°C while stirring. In this case, the
15 surface-treated particles may be filtered after reaction.
[0023]

The insulating conductive particle of the present invention can be preferably used as a conductive particle in an anisotropic conductive adhesive. Such an
20 anisotropic conductive adhesive can be manufactured by uniformly mixing the insulation coated conductive particles with an insulating adhesive, serving as an adhesive component, in the usual manner, together with an organic solvent or inorganic filler if necessary. This
25 anisotropic conductive adhesive can be formed to be a

paste or a film in the usual manner.

[0024]

When the amount of the insulation coated conductive particles blended in the anisotropic conductive adhesive is too small, the conduction reliability may deteriorate. On the other hand, when the blended amount of the insulation coated conductive particles is too large, the insulating properties may be insufficient. Therefore, it is preferable that the blended amount of the insulation coated conductive particles be in the range from 1 to 20 vol%.

[0025]

Known insulating adhesives can be used as the insulating adhesive to be used in the above anisotropic conductive adhesive. Examples of the insulating adhesive include: thermosetting liquid insulating adhesives comprising a polymerizable component such as liquid epoxy resin and a curing component such as an imidazole-based curing agent or a modified amine-based curing agent; liquid insulating adhesives comprising acrylate-based resins having a polymerizable double bond in conjunction with a curing catalyst; and liquid rubber-based adhesives comprising a thermoplastic resin such as acrylic resin, a rubber-based resin such as SBR, SIS, and polyurethane, and the like. Especially, even in a case where an insulating

resin having a carboxyl group is used as the insulating resin of the insulation coated conductive particle, and epoxy resin is used as the insulating adhesive, the storage stability is good because a region of the
5 insulating resin in the vicinity of its surface is cross-linked by the polyfunctional aziridine compound.

[0026]

Various additives such as a thickener and a surface active agent may be added to the anisotropic conductive
10 adhesive, if necessary.

[0027]

The insulation coated conductive particle of the present invention can be preferably used when various electronic parts are bonded to a wiring board.
15

EXAMPLES

[0028]

The present invention is more specifically described below, based on several examples.

20 [0029]

Comparative Example 1

The insulation coated conductive particle of Comparative Example 1 was obtained by coating the surface of a conductive particle (AU204, Sekisui Chemical Co.,
25 Ltd.) - in which an Ni-AU electroless plating layer was

formed on the surface of a styrene-based resin particle having a diameter of 4 μm - with a styrene-acrylic acid copolymer (PP-2000S, Dainippon Ink and Chemicals Inc.), having a thickness of 0.2 μm , in the usual manner.

5 [0030]

Example 1

A solution obtained by dissolving 5 parts by weight of trimethylolpropane-tri- β -aziridinypropionate (TAZM) in 95 parts by weight of ethanol was evenly sprayed onto the
10 insulation coated conductive particle of Comparative Example 1, and was then heated and dried at a temperature of 100°C so as to cause cross-linking reaction. In this manner, the insulation coated conductive particle of Example 1 was obtained.

15 [0031]

Example 2

100 parts by weight of the insulation coated conductive particles obtained in Comparative Example 1 were dispersed in 100 parts by weight of ethanol. 2 parts
20 by weight of trimethylolpropane-tri- β -aziridinypropionate (TAZM) was added to the above dispersion liquid and was dispersed by stirring the liquid. Then, the dispersion liquid was heated and stirred at a temperature of 65°C for 4 hours so as to cause cross-linking reaction. The
25 dispersion liquid after cross-linking was filtered and was

then dried at a temperature of 80°C for 30 minutes. In this manner, the insulation coated conductive particle of Example 2 was obtained.

[0032]

5 Example 3

The insulation coated conductive particle of Example 3 was obtained in the same manner as that in Example 2, except that trimethylolpropane-tri- β -aziridinypropionate (TAZM) was replaced with tetramethylolmethane-tri- β -
10 aziridinypropionate (TAZO).

[0033]

Example 4

The insulation coated conductive particle of Example 4 was obtained in the same manner as that in Example 2,
15 except that trimethylolpropane-tri- β -aziridinypropionate (TAZM) was replaced with N,N-hexamethylene-1,6-bis-1-aziridinecarboxamide (HDU).

[0034]

(Evaluation)

20 10 parts by weight of the insulation coated conductive particles of each of Comparative Example 1 and Examples 1 to 4 were individually put into 90 wt% of three types of solution, i.e., toluene, MEK, and ethyl acetate. Then the thus obtained mixture was left at a room
25 temperature for 100 hours so as to allow the insulation

coated conductive particles to settle out. Next, supernatant liquid was separated. The thus obtained supernatant liquid was heated to remove volatile components and thereafter weight of non-volatile components was measured. The weight of the non-volatile components corresponds to the weight of the insulating resin dissolved in the solvent. Table 1 shows the ratio (wt%) of the portion of insulating resin that was dissolved in the solvent compared to the entire amount of insulating resin.

[0035]

Moreover, the insulation coated conductive particles that settled out were dried. The thus dried insulation coated conductive particles were placed between a pair of copper electrodes ($\phi 6$ mm \times 125 μ m) and a voltage was applied across the electrodes. The voltage at which leak occurred (withstand voltage) was measured. The measurement results are shown in Table 1.

[0036]

In addition, an adhesive composition containing 35 parts by weight of phenoxy resin (YP50, Tohto Kasei Co., Ltd.); 30 parts by weight of epoxy resin (YL980, Japan Epoxy Resins Co., Ltd.; equivalent weight of epoxy: 185 g/eq); 35 parts by weight of imidazole-based curing agent containing epoxy resin dispersed therein (HX3941HP, Asahi

Kasei Corporation); 20 parts by weight of conductive particles (conductive particles of any of Examples 1 to 4 and Comparative Example 1); 40 parts by weight of toluene; and 40 parts by weight of ethyl acetate was applied onto a polyethylene terephthalate film for which a peeling treatment was performed so as to have a thickness of 25 μm in the dry state. Then, the adhesive composition was dried at a temperature of 80°C for 5 minutes to form an adhesive layer. In this manner, an adhesive sheet was formed. Subsequently, an insulating TEG for evaluating a short circuit (chip size: 25 \times 2.5 mm; the number of bumps: 8376; bump size: 35 \times 55 μm ; space between bumps: 10 μm) that included an ITO wiring arrangement arranged in the form of the teeth of a comb on a glass substrate, was pressure-bonded to an adhesive-layer surface of the adhesive sheet. This was achieved by means of a bonder under conditions in which the attained temperature was 210°C and the pressure-bonding time was 10 seconds. Then, the insulation resistance between the bumps was measured and the rate of occurrence of a short circuit was calculated. The measurement results are shown in Table 1.

[0037]

[Table 1]

Solvent	Evaluation items	Comparative Example 1	Example			
			1	2	3	4
Toluene	Ratio of dissolved portion (wt%)	26.9	7.5	3.8	4.0	8.3
	Withstand voltage (kV)	0.4	2.0	2.4	2.3	1.8
MEK	Ratio of dissolved portion (wt%)	27.4	8.2	4.4	6.1	8.7
	Withstand voltage (kV)	0.5	1.9	2.2	2.0	1.5
Ethyl acetate	Ratio of dissolved portion (wt%)	26.6	7.2	4.0	5.8	7.9
	Withstand voltage (kV)	0.6	2.1	2.6	2.1	1.6
Rate of occurrence of short circuit (%)		6.2	0.3	0.0	0.0	0.5

[0038]

5 As is apparent from Table 1, the insulation coated
conductive particles of Examples 1 to 4 are better in
terms of both a solvent resistance and a withstand voltage
property for all of the above solvents, as compared with
the insulation coated conductive particles of Comparative
10 Example 1 that were not surface-treated with an aziridine
compound. Therefore, the conduction reliability is also
improved. Moreover, the rate of occurrence of a short
circuit is very small and the favorable storage stability
is expected.

15

INDUSTRIAL APPLICABILITY

[0039]

The insulation coated conductive particles of the present invention have excellent a solvent resistance, an excellent withstand voltage property, and improved conduction reliability. Moreover, the rate of occurrence
5 of a short circuit is very small and it is therefore expected that storage stability of the insulation coated conductive particle of the present invention is favorable. Accordingly, the insulation coated conductive particles of the present invention are useful as conductive particles
10 in the manufacture of anisotropic conductive adhesives.